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^{3}He Recovery from a Tritium-aged LANA75 Sample

K. L. Shanahan

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Savannah River National Laboratory
Savannah River Nuclear Solutions, LLC
Aiken, SC 29808

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AUTHOR:

K.L. Shanahan, Hydrogen Processing Group Date

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G. A. Morgan, Hydrogen Processing Group Date

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Kirk L. Shanahan

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Introduction

^{3}He recovery is a topic of recent interest. One potential recovery source is from metal hydride materials once used to store tritium, as the decay product, ^{3}He, is primarily trapped in the metal lattice, usually in bubbles, with such materials. In 2001, a Tritium Exposure Program (TEP) sample known as LANA75_SP1 was retired and the material was removed from the test cell and stored. Subsequently scoping temperature programmed desorption (TPD) experiments were conducted on that material to see what it might take to drive out He and residual H isotopes (the heel). Two experiments consisted of heating the sample in the presence of an excess of tin (the so-called Sn fusion experiment), and one was a simple TPD with no additives.

Prior data [1] on the so-called '21-month bed' material in the 1980's had produced ~21 cc of gas per gram of a LANA30 material ($LaNi_{4.7}Al_{0.3}$), with approximately 67% of that being ^{3}He and the rest being D_2 (Fig.3). However, the material had to be heated in excess of 850 ^oC to obtain that level. Heating to less produced approximately half that amount of gas. The data also showed that ^{3}He was released at different temperatures than the residual hydrogen isotopes. Unfortunately this implies full ^{3}He recovery will be a difficult process. Therefore, it seemed advisable to attempt to extract as much information from the 3 scoping experiments from 2001-2 as possible.

Experimental

Two types of experiments were run, a simple TPD on the material, and two attempts at what was known as tin fusions, wherein an excess of metallic tin (Sn) was added to the sample and allowed to melt during heating (Sn m.p. 233 ^oC). Ostensibly, the Sn would alloy with the LANA material and thereby stimulate entrapped gas release. All three experiments consisted of heating the sample to a given temperature and holding for short times. The gases evolved were then sampled and the sample cooled. Subsequently, the sample was heated again, sometimes to higher temperatures, and again grab samples of the evolved gases were taken and analyzed. The Sn fusions did not produce noticeably different results from the simple TPD.

The experimental assembly used was a variation on the standard 'grab sampling assembly' (GSA). In both cases, the LANA material resided in a sample cell which was closed with a double Nupro valve fitted with Cajon 4-VCR connections. The sample cell and valve assembly is typically referred to as a test cell. For standard TEP test cells, the sample cell simply held powder or foil forms of the hydride material. For the Sn fusions and the one TPD experiment, the hydride material was placed in a glass tube with one closed end, and the tube was placed in a Nupro sample cell made from standard ½" OD tubing capped at one end and fitted with an 8-VCR connection on the other. A 4-VCR to

8-VCR adapter was used then to connect to the double valves. The GSA was then formed by connecting a 4-VCR cross to the tritium manifold, connecting a pressure sensor on one port of the cross, a 25 cc gas sample bomb (also double valved) on another, and the test cell on the third port. During grab sampling, only the valves to the test cell were open which minimizes the attached volume and disturbs the sample the least when obtaining gas samples. The Sn fusions and TPD also followed this procedure. At room temperature, the volume of the test cell plus connecting valves, pressure sensor, and cross (up to the closed manifold and sample bomb valves) was 28.88 cc. The volume of the test cell only (past both of the double valves) was 19.46 cc.

Since these were scoping studies only, no volumes were determined with the sample at temperature in 2001. Heating the sample introduces a temperature gradient in the test cell, and the use of the nominal room or sample temperature in the Ideal Gas Law (IGL) equation used to compute mass balance is incorrect. While one can attempt to compute an appropriate average temperature via heat transfer modeling, the far simpler method is to determine the apparent volume with the sample at temperature, and use that volume in the IGL with the nominal manifold temperature as T for data obtained with the sample at a given temperature (of course V now depends on sample T). In order to compute the mass balances as reported herein, some estimates of the apparent volumes at temperature were required.

This was done by assembling a mock GSA which simply capped the cross port where the pressure transducer normally went, and placed a valve where the sample bomb usually went. The same type Nupro tube sample cell (empty) and double valve assembly was used as the one used in the original Sn fusion/TPD experiments. This produced a system volume of 22.88 cc at room temperature, which is comparable to that obtained in the actual experiments. The empty cell was then heated with the same type of heater to several temperatures used in the 2001 experiments, and the pressure recorded. From that the apparent volume was calculated, and from that the (negative) change in volume from room temperature was computed. This change was then added to the 28.88 cc volume from the original experiments to estimate the volume at temperature for the 2001 runs. These adjusted volumes were used in the mass balance calculations. It is assumed that this simulation was adequate since only the lowest 2-3” of the 8-1/2” sample cell was contained in the heater. Thus the primary region of temperature gradient would be from the top of the heated region approximately up to the VCR adapter, which was identical to that in the 2001 experiments. In other words the extra volume obtained in 2001 was assumed to be at the nominal manifold temperature and not affected by the heater.

The LANA75_SP1 sample contained a LANA75 material that was of very low secondary phase content, i.e. single phase (thus the ‘SP’). The LANA material was passivated at room temperature on 2/8/2001 and the passivation gas was grab sampled. The results showed no He to note and only N_2 and O_2. The concern at the time was that the passivation process would release some of the trapped gases, but recent work has shown that was unlikely [2], which is confirmed by these analytical results.

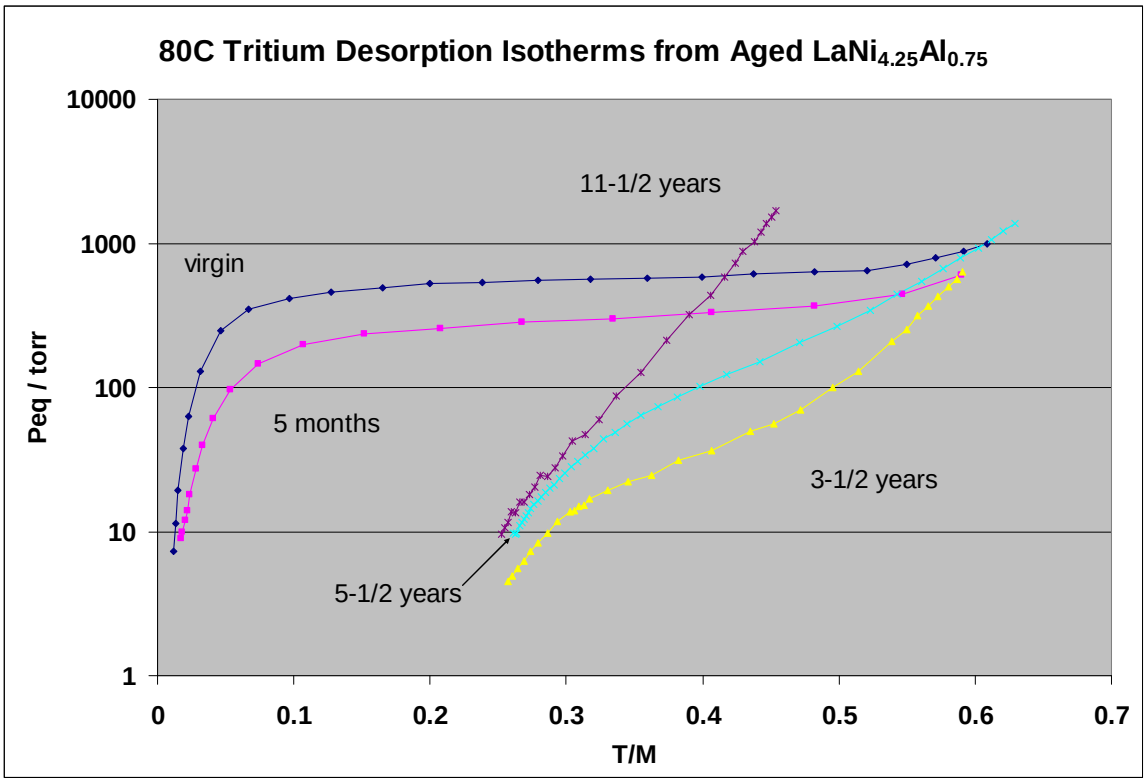
The sample had seen approximately 12 years of tritium exposure. Of course some of that time occurred during isotherm determinations. The material had developed a large hydrogen isotopes heel (as expected) which was ~0.25 Q/M units (Q = H + D + T). Note that this sample had had at least 8 D_2 exchanges/isotherms determined on it prior to retirement, and had a very low residual T content in the heel. This hydrogen gas is also expected to be driven out during the TPD experiment. The final He/M ratio obtained was estimated, as described below, to be 0.30 He/M units. Thus if all the gases held in the material could be thermally driven out, ~140 cc gas /gm LANA would be expected, which would produce a pressure of ~1500-2000 torr in the GSA, depending on cell temperature and sample weight. This high of a pressure was never observed in any single experiment. However, because two heats were conducted for each sample, that pressure may have been split between heats and thus must be computed.

All the experiments were conducted by placing about 0.345-0.346 grams of material in a glass tube closed at one end (essentially a narrow glass test tube) which would fit inside the Nupro sample cell. Double valve assemblies were then attached. The test cell was then mounted on the GSA. The bottom end of the tube was heated with the standard Glas-col 150cc beaker heater in use at that time. Temperatures were measured by clamping thermocouples to the tube end. Data collection was manual and recorded in the lab notebook WSRC-NB-98-00255.

LANA75_SP1 Loading History and ^{3}He/M Ratio Estimation

There are a variety of factors that must be considered in estimating the He/M ratio of a sample. Clearly, the tritium content as a function of time is the primary concern. This is relatively easy to estimate during periods of sample storage. One minor complication encountered there is the effect of adsorbing T from the gas overpressure left at the end of loading when the T in the bulk metal hydride decays. With no T-decay effects, it would be relatively simple to use the determined isotherms to estimate the equilibrium points. However, tritium aging induces plateau pressure shifts and heel growth, which makes estimation difficult without a complete series of aged material isotherms or some other way of compensating for these effects. (Changes in T isotherms from LANA75 material with aging time are shown in Figure 1.) Then it is routinely observed that a small percentage (3-5%) of the decay ^{3}He escapes to the gas phase. This is thought to be due to He being born within a diffusion length of the surface and fortuitously diffusing out of the material. This makes the surface-to-volume ration of the test material important. When dynamically probing the material by determining isotherms, cycling, or exchanging isotopes, the problem becomes somewhat more difficult. In theory, with an exact record of what was done to the sample, a computer model could accurately predict the obtained He/M ratio. However, no such model is available at this time, so we will herein make estimates of these factors.

Figure 1. 80 °C Tritium Isotherms from T-Aged LANA75



LANA75_SP1 Loading History

The following history is reconstructed from two notebooks. Entries from 1987 to 1998 were recorded in a green RECORD book with white stenciled label of "LANA.75 SP1" on the front and "LANA75 SP1" in black marker on the spine. In October of 1998, the notebook WSRC-NB-98-00255 was acquired to record the remainder of experimental results on LANA75_SP1. "LANA.75 SP1" is written on the front cover label of that notebook, and "LANA75 SP1" is written on the spine of the notebook.

LANA75_SP1 (hereafter referred to as SP1) was assembled on 3/25/1987 from a special lot of material (Ergenics 1304-B-2). The material contained very little secondary phases, and was therefore called a 'single phase' material, which is the genesis of the 'SP' in the sample name. 5.32 gr. of material was placed in the sample cell. It was D_2 activated and transferred to the Material Test Facility (MTF) in H-Area for tritium studies.

It was first loaded with tritium of 97.371% purity on 4/15/87, and was left with a T_2 overpressure of 201 torr, giving an initial T/M of 0.586. It was stored until 8/12/87 when an 80 °C tritium desorption isotherm was determined, which was finished on 8/14/87. It was reloaded on 8/17/87 with 95.707 % tritium to an initial T/M of 0.658, with a residual overpressure of 200 torr, and placed in storage until 6/15/88. Another 80 °C desorption isotherm was initiated then, which was finished on 6/29/88. It was immediately reloaded

and a second isotherm determined, which finished on 7/13/88. It was reloaded with 98.051% T_2 on 7/18/88 with a residual overpressure of 1096 torr, giving an initial T/M of 0.619. Beginning 7/29/88, it was then cycled to 150-160 ^oC 20 times, and subsequently evacuated and baked out at 150 ^oC (final pressure was ~350 microns). On 8/11/88, it was reloaded with 98.161% T_2 and an 80 ^oC desorption isotherm then determined, which was finished on 9/14/88 with a 150 ^oC bakeout. On 9/16/88, it was reloaded with tritium of unknown purity and placed in storage until12/5/88. On that date another 80 ^oC tritium desorption isotherm was started, which was finished on 1/6/89 with a 150 ^oC bakeout. It was reloaded on 1/18/89 with tritium of unknown purity and left with a residual pressure of 1577 torr. It was then left in storage until October of 1998.

On 10/6/98, a grab sample of the cell overpressure was taken, and was followed on 10/13/98 by initiating an 80 ^oC desorption isotherm. Due to an operator error, this isotherm was terminated early and followed by a short bakeout. It was reloaded with tritium on 10/23/98, and followed by another 80 ^oC desorption isotherm, which was completed on 11/4/98. On 11/11/98, the sample was reloaded as part of a desorption isotherm (one-shot load), and a second 80 ^oC desorption isotherm determined, which was completed on 11/30/98. Following that, seven deuterium heel exchanges were performed on the sample during the period of 12/2/98 to 3/9/99. Then an 80 ^oC D_2 desorption isotherm was measured, which took from 3/11/99 to 3/16/99. Subsequently, the temperature was raised to 150 ^oC and several more points taken, ending on 3/24/99. Duplicate grab samples were taken during the seventh D_2 heel exchange experiment, and analyses indicated 1.496 and 1.502% T content. Grab samples were also taken twice during the first D_2 isotherm on sequential cycles and indicated 0.924% and 0.625% T content respectively. These deuterium experiments effectively end the tritium exposure for LANA75_SP1. The total number of elapsed days between 4/15/87 and 11/30/98 is 4247 days (11.6 years).

The sample was then stored until 10/5/99 when it was grab sampled again. Subsequent to the 150 ^oC partial desorption isotherm of 3/16/99, a volume calibration had been performed on the test cell which included determining the sample cell volume, i.e. the volume calibration gas used (Ar) was admitted to contact the hydride material. This procedure is always finished by evacuating all parts of the apparatus touched by the Ar to < 50 microns, so the sample cell should not have had any pressure in it on 10/5/99, unless He release had occurred. However, a pressure of 121 torr was observed in the grab sample apparatus on 10/5/99, and subsequent analysis showed it consisted of 4.089% tritium, 0.38% deuterium, and 0.064% protium, 77.064% ^3He, 17.688% nitrogen, 0.786% Ar, and traces of oxygen, CO_2, and methanes. The N_2 present indicated that an air leak had occurred, probably through the valve seats, suggesting the sample may have reacted with atmospheric O_2. Recent work has shown that no significant damage to the sample should have occurred [2]. The ^3He concentration suggested that some He release had occurred as well, although whether it was stimulated by reaction with atmospheric oxygen or not is unknown. The T_2 concentration of >4% is inconsistent with the prior T content determinations if the gas came from the sample, however, recently W. Shmayda has shown that exposing T contaminated surfaces to humid air can produce a tritium exchange and subsequent release[3]. Thus, the T detected in the sample likely was

displaced from the cell walls as opposed to coming from the sample itself. Subsequent to taking the grab sample, another D_2 heel exchange was conducted on 10/6,7/99, and followed by an 80 ^oC D_2 desorption isotherm determination, which ended on 12/1/99. On 1/19/2000, the sample was baked out at 150 ^oC. Another volume calibration was performed on the sample on 1/25/00 (final pressure was <50 microns). On 2/8/01, the sample was passivated. Analysis of the grab sample taken during the passivation procedure showed no noticeable ^3He, suggesting that the air leak detected in the 10/5/99 sample was not the cause of the observed ^3He. On 7/6/01, the test cell was disassembled and the hydride material removed and placed in a glass jar for subsequent experimentation (Sn fusions and TPDs).

$^3He/M$ Ratio Estimation

He accumulation is estimated by considering two types of time periods, that where static storage is ongoing, and that where dynamic processing is ongoing. The dynamic processing is broken down further into two parts, isotherm determination and cycling. The He accumulation during static storage is relatively easy to compute via simple radioactive decay. There were six such periods, and Table 1 lists them, showing starting date, length of storage, and accumulated He in He/M ratio units.

Table 1. Static Storage He Accumulation Periods

Date	Days Stored	He/M accumulated
4/15/87	119	.0075
8/17/87	303	.021
7/18/88	11	.0007
8/11/88	20	.0015
9/16/88	80	.0060
1/18/89	3555	.2272
Total	4088	.2639

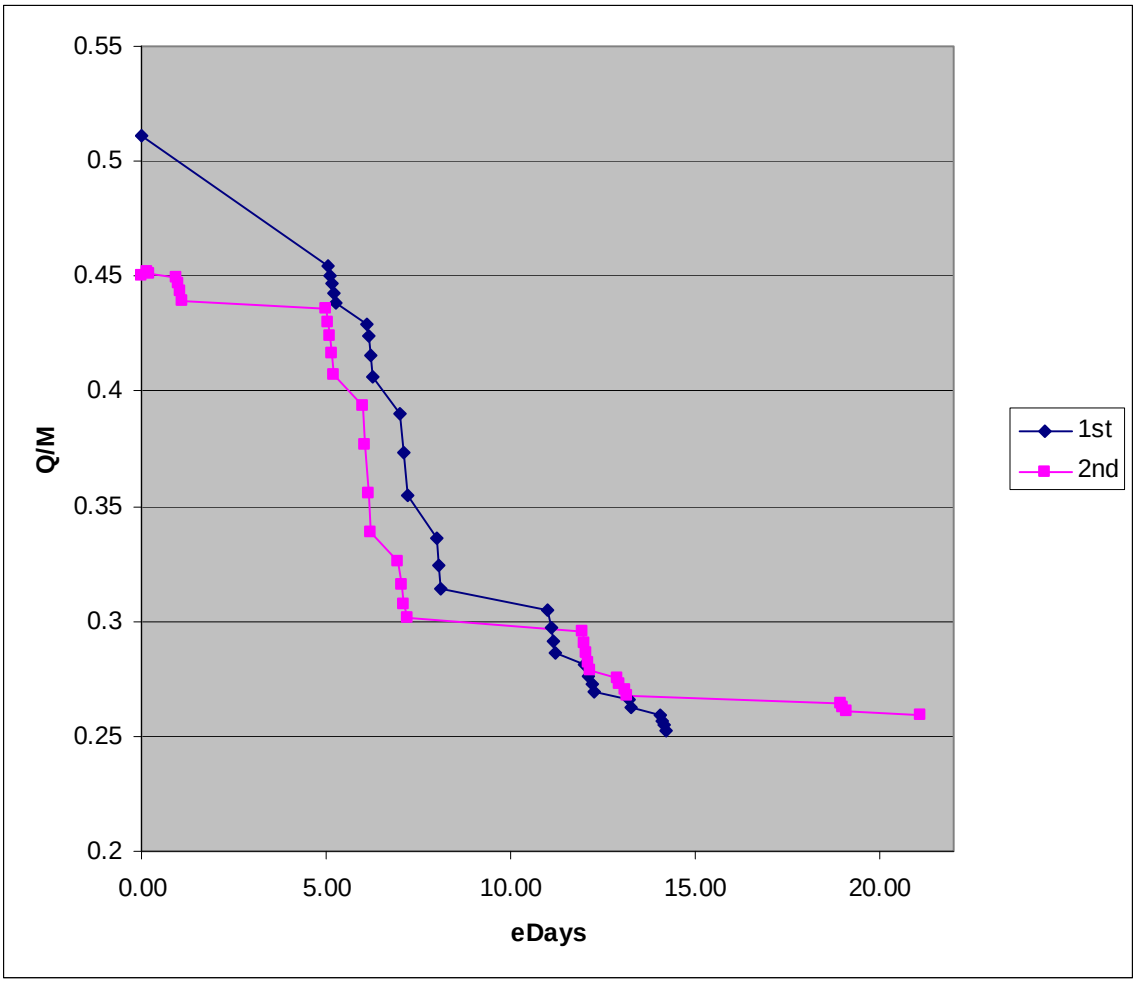
This calculation assumed the initial T/M determined from the loading data and $\%T_2$ purity numbers, which required assuming 98% T_2 purities for the loads of the last two decay periods. In addition the impact of adsorbing T_2 from the cell overpressure as the solid phase T decomposes must be considered. The moles of tritium in the gas at the start of the decay period can be directly calculated from the Ideal Gas Law. That can be converted to an equivalent addition to the T/M value assuming it would all be adsorbed. The sum of those numbers came to ~0.1 He/M units, but that assumed there was enough time to decay enough bulk T to promote the full absorption of the gaseous T. For the short time decay periods this is not true. Therefore the Q/M values for the material during decay period 6 was computed as a function of time, assuming no refreshing, and it was found that ~200 days were required to deplete the bulk to an equivalent level of the gaseous T. Thus shorter time frames would absorb proportionately less, and the additional He/M amount computed by full decay of the gas phase T (absorbed into the

solid) was scaled by the fraction of 200 days the sample was decayed, reducing the He/M addition proportionately. This is a simplistic assumption of course, but this process showed that the only significant decay periods were the last two. The fifth decay period added 0.007 He/M units, and the sixth added 0.031 in this approximation, for a total of 0.038. Then the only other correction that might be applicable is to subtract the He that would have been formed in the gas phase during the time when T_2 was present there. Decay calculation for a 200 day period for the given initial overpressures showed this was an insignificant correction. Therefore the correction to the final sample He/M ratio from refreshing the bulk T from the gas phase is estimated as +0.038.

The next problem was to estimate the He/M addition obtained during the dynamic processing events. Q/M values were available for the two isotherms determined in late 1998. Plots of Q/M vs. time were constructed for the two isotherms, and are shown in Figure 2. Of note is the fact that the heel of 0.25 Q/M was always present and contributing to the he accumulation. This makes these isotherms the most severe case. He accumulation was estimated by assuming a single Q/M value for those periods where the Q/M values changed little. During the times when Q/M changed significantly, the average Q/M value for that period was used in the decay calculations. This procedure gave accumulated He/M units of 0.0006 and 0.0008, respectively, which are insignificant alone. However, during the lifetime of SP1, seven isotherms were determined and two periods of cycling were conducted. Counting the cycling periods as one isotherm and using the average accumulated He/M, the total accumulated He/M would be 8*.0007 = .006 rounded up. This should be added to the other dynamic calculation and the static result for an estimated He/M ratio in the sample of 0.264 + 0.038 + 0.006 = 0.308. The difference between the nominal sample total tritium exposure period (4247 days) and the total static storage period (4088) gives an average time per 'isotherm' of ~20 days, which is consistent with the total isotherm determination times shown in Figure 2.

The final correction to this estimate should be for the fraction of the He born in the solid that manages to escape via diffusion to the gas phase. No information on this was recorded prior to the 1998 work. The grab sampling of 10/6/98 showed the gas was 99.755% ^{3}He, which is expected since the tritium heel and long storage period insured that all the hydrogen isotopes were in the solid. The GSA volume of 25.30 cc and the observed pressure in that volume of 193.58 torr gives the He present as 0.268 mmoles. This represents a He/M ratio of 0.0034. As the product of the longest single storage period, which constituted ~85% of the total lifetime of the sample, He losses to the gas phase during the remainder of the storage period will be considered to not exceed 0.003. Thus a maximum reduction in He/M ratio of ~0.006-0.007 should be applied. Rounding off for simplicity gives a final estimate for the sample He/M ratio of 0.30. This is obviously a very rough estimate, but given the large number of unknowns used in this estimate, it represents about the best one can do at this point without detailed modeling. One way to have cross-checked this was to have Brian Oliver of PNNL completely desorb a small sample of the LANA material, and directly compute the He/M and Q/M ratios, but this was not done.

Figure 2. Q/M values for two isotherms from late 1998 as functions of time



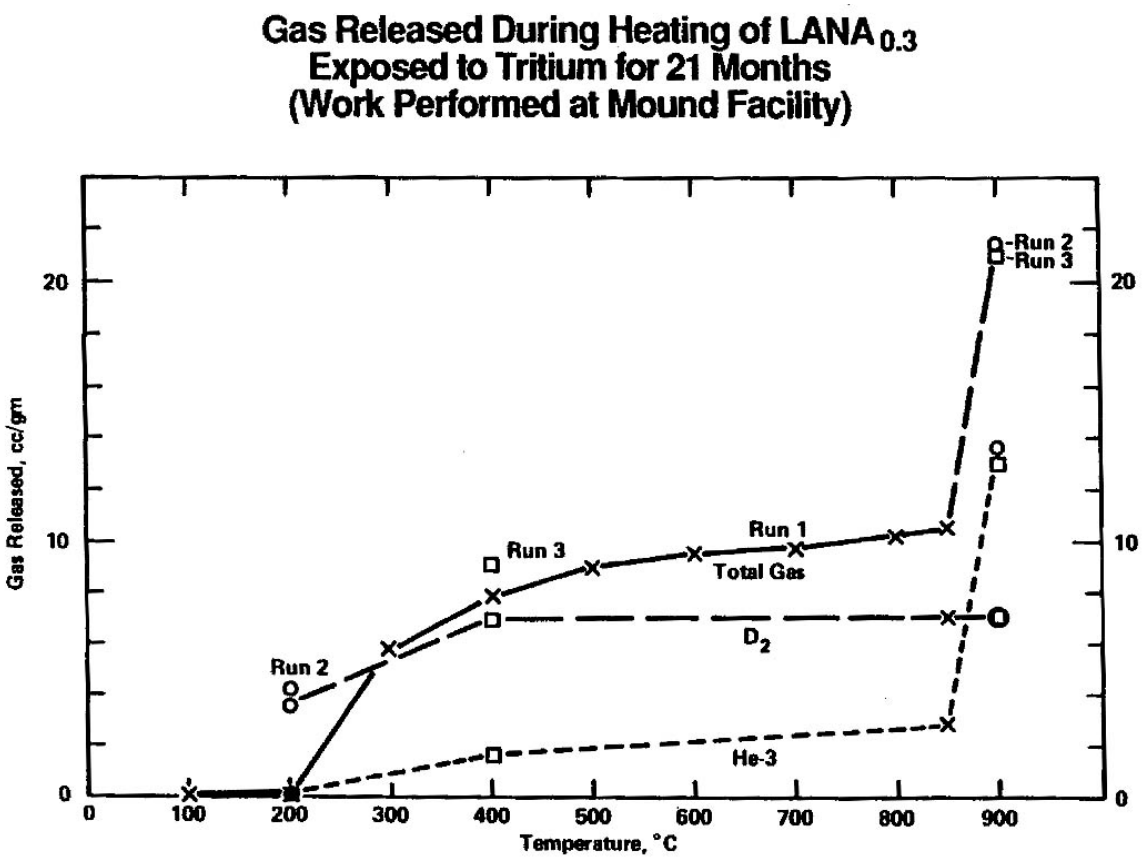
Theoretical Amount of Gas Release

Given the estimated He/M ratio of 0.30 and measured Q/M ratio of 0.25, the amount of gas released from a gram of the SP1 material can be calculated. One gram of SP1, with a molecular weight of 408.57 g/mole, contains 0.00245 moles of LANA, or 0.0147 moles of 'M' (6 M per formula unit of LANA). The amount of He in that quantity of LANA would then be 0.00441 moles, and the amount of Q2 would be 0.00184 moles. These translate to 0.0073 grams of D_2 (assuming the Q_2 is primarily D_2) and 0.0132 grams of ^{3}He, giving a total weight of 1.0205 grams. The standard cubic centimeters of gas represented by the total of .00625 moles of gas is 140 scc, which gives an expected maximum amount of gas released per gram of aged SP1 material equal to 140/1.0205 = 137 cc/gram.

In 1988, LANA.3 or LANA30 material, from a hydride bed exposed to T for 21 months was examined by Mound personnel and subsequently reported on by R. T. Walters in a

presentation (DPST-88-498). Slide 17 of that presentation is reproduced below as Figure 3. These results show a total gas amount released of ~ 21 cc/g. Twenty-one months is roughly 639 days, which is ~15% of 4247 days. Fifteen percent of 137 cc/g is ~20.5 cc/g, which is excellent agreement. Figure 3 also shows that at ~850 ^{o}C, only about half of the final total gas released has actually been driven off, with only slightly less having come off at ~400 ^{o}C. Further, heating to ~900 ^{o}C released primarily only ^{3}He. Below, we will compute the amount driven off from SP1 at several different temperatures for comparison.

Figure 3. Slide 17 from 1988 Presentation. Gas volumes driven off of a T-aged LANA30 material.



Results and Discussion

Simple TPD (no Sn)

A 0.346 gram SP1 sample was heated to ~450 ^{o}C and held there for ~160 min. Figure 4 shows the pressure and temperature time profiles. Pressure started increasing very early in the run, but there appears to be some preheating that was not recorded. Of note is that there is considerable pressure developed at 450 ^{o}C, and that it seems to be continuing to develop even after nearly 2 hours of experiment time. The abrupt pressure drop at the

end is due to grab sampling. The sample showed 83.65% ^{3}He, 2.42% H, 13.76% D, 0.042% T.

Subsequently that sample was cooled down and the next day the sample cell was reopened to the evacuated grab sample apparatus. It was then heated to 650 ^{o}C and held for ~3 hours. The P, T time profiles are shown in Fig. 5. Again, the abrupt pressure drop at ~215 minutes was due to grab sampling. Subsequently the sample was sequentially reduced in P in a controlled fashion that could yield mass information if analyzed. The initial pressure jump is due to residual P in the test cell from the day before, which was 83+% ^{3}He. The grab sample analyzed out as 78.82% ^{3}He, 3.76% H, 17.076% D, 0.207% T, so the second heat appears to have driven out more H isotopes than He.

Sn Fusions

Two tin fusion experiments were run, and both had two parts (heats). The first experiment used 0.345 g of LANA75 material in the test apparatus with an addition of 1.439 g Sn, and the second used 0.345 g of LANA and 3.377 g Sn.

The first Sn fusion was initially heated only to about 350-360 ^{o}C, but significant pressure was developed. P and T profiles are in Fig. 6. Of note is the failure to get a P increase until the T reached ~300 ^{o}C. The grab sample taken at the run's end showed 73.78% ^{3}He, 2.60% H, 23.29% D, and 0.042% T. The sample was then allowed to cool.

The next day the test cell was again opened to the GSA, and it was noted that the P seemed to be increasing, not stable, so a grab sample of that was taken. It showed 79.81% He, a slight increase. No indications of N_2 or O_2 were observed in the mass spectrum suggesting no leak had occurred. Apparently the sample had continued to offgas at room T. The sample was then evacuated which implies that the subsequent heating of this sample should not show a pressure jump upon opening the sample cell to the GSA.

After a 4 month break, the second heat on this sample was attempted and results are shown in Fig. 7. There was a slight P jump (~5 torr) when the test cell was first opened. It is not clear if this is evidence of continuing He release at room temperature. The sample was heated up to ~475 ^{o}C and additional P increase was observed. The grab sample was lost due to valving error.

A second sample was prepared for Sn fusion as described above. It was heated to ~450 ^{o}C and held for ~250 minutes (Fig. 8). The end state grab sample showed 80.69% ^{3}He, 3.29% H, 15.715% D, 0.044% T.

Over a month later, the second heat to ~475 ^{o}C was performed (Fig. 9). Some additional pressure increase was obtained over the initial jump due to residual gas in the test cell. . Grab sampling showed 75.08% ^{3}He, 4.75% H, 19.96% D, and 0.05% T. This may indicate that the pressure increase observed in this heating was only desorption of readsorbed hydrogen isotopes.

Computation of %Theoretical Gas Release

Computation of the cubic centimeters of gas released is straightforward for the first heats of these samples. In the second heats, the 1st heat residual in the test cell must be subtracted to determine how much additional gas was driven off in the 2nd heating, except for the 1st Sn fusion experiment, where the sample cell was emptied before the 2nd heat. These calculations indicate that ~59% of theoretical was released in the first Sn fusion run, ~68% in the 2nd Sn fusion run, and ~75% in the simple TPD. This is probably a function of the maximum temperature attained and time at temperature in the runs. Longer times and higher temperatures were used in the simple TPD, and the 2nd Sn fusion used longer times and slightly higher temperatures than the first Sn fusion. Mass spectral results however, indicate that the He/Q ratio is higher (~3) than in the 1988 Mound data (~2). But with significantly less aging time, which would give both lower He and Q heel content, this also may have been anticipated. With the longer aging time on the SP1 material, the Q heel would have maximized while the He content continued to grow, displacing the He/Q ratio to higher values with time.

Conclusions

Significantly more gas is released at temperatures lower than 8-900 ^{o}C, which contrasts with the 1988 Mound data, but 100% release is not obtained at the temperatures attained, which is also consistent with the Mound data. The higher gas volume released is consistent with the longer aging time of the LANA7_SP1 sample (11.6 years) vs. the 21-month bed material (1.75 yr). Studies on T-aged Ti indicate that as the He/M ratio increases the onset of desorption lowers [4]. Similar behavior in the LANA would also lead to higher releases in the SP1 material at the temperatures attained in the reported studies. It should also be noted that the Sn fusion approach does not seem to offer any particular advantage to ^{3}He removal.

Acknowledgements

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Figure 4. Pressure and Temperature profiles from 1st heating of LANA75_SP1

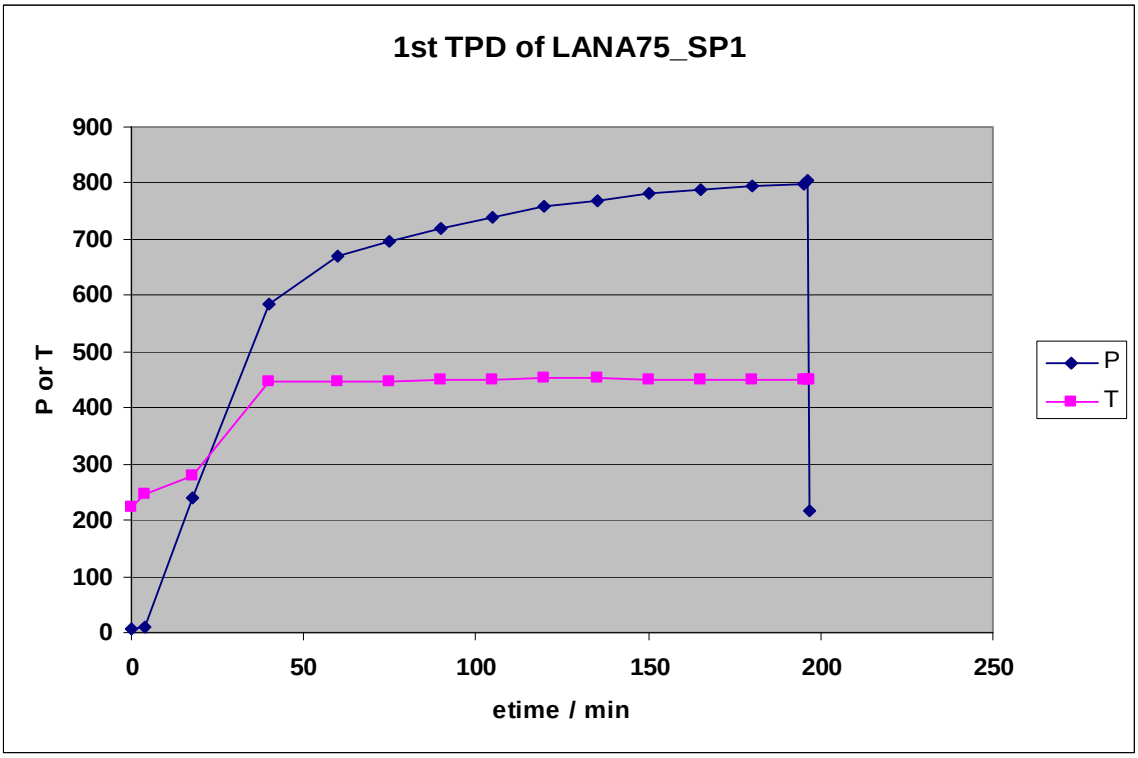

Figure 5. Pressure and Temperature profiles from 2nd heating of LANA75_SP1

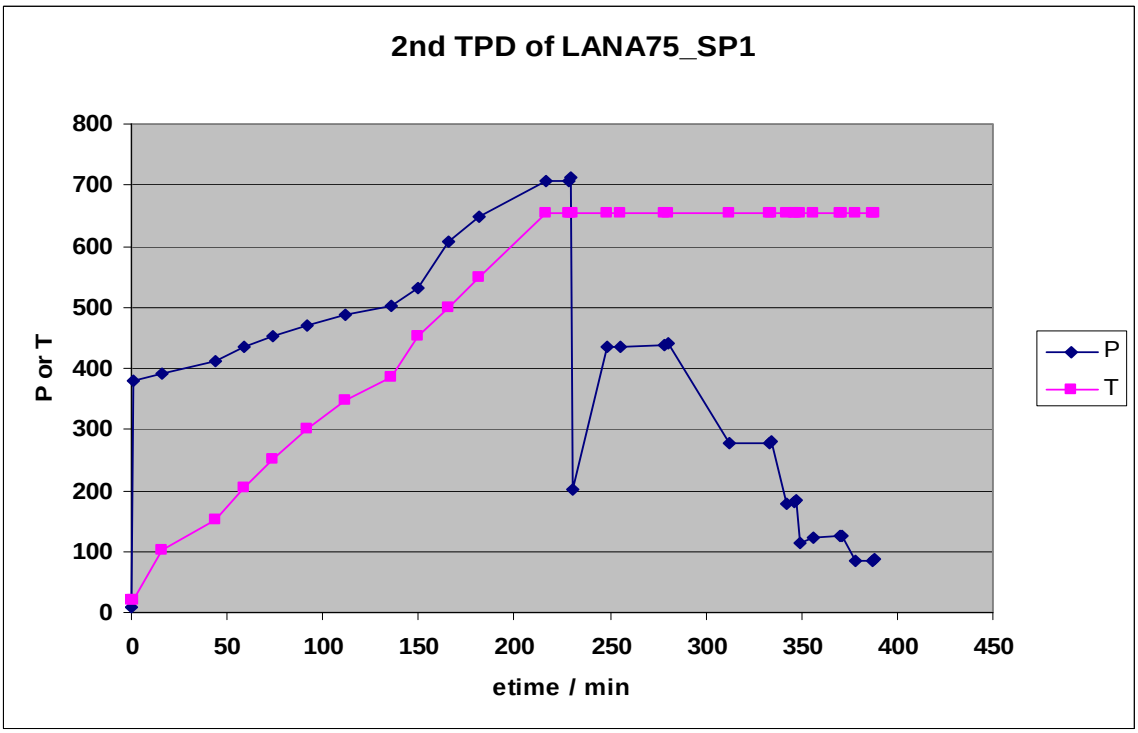

Figure 6. Pressure and Temperature profiles from 1st heating of 1st Sn fusion sample

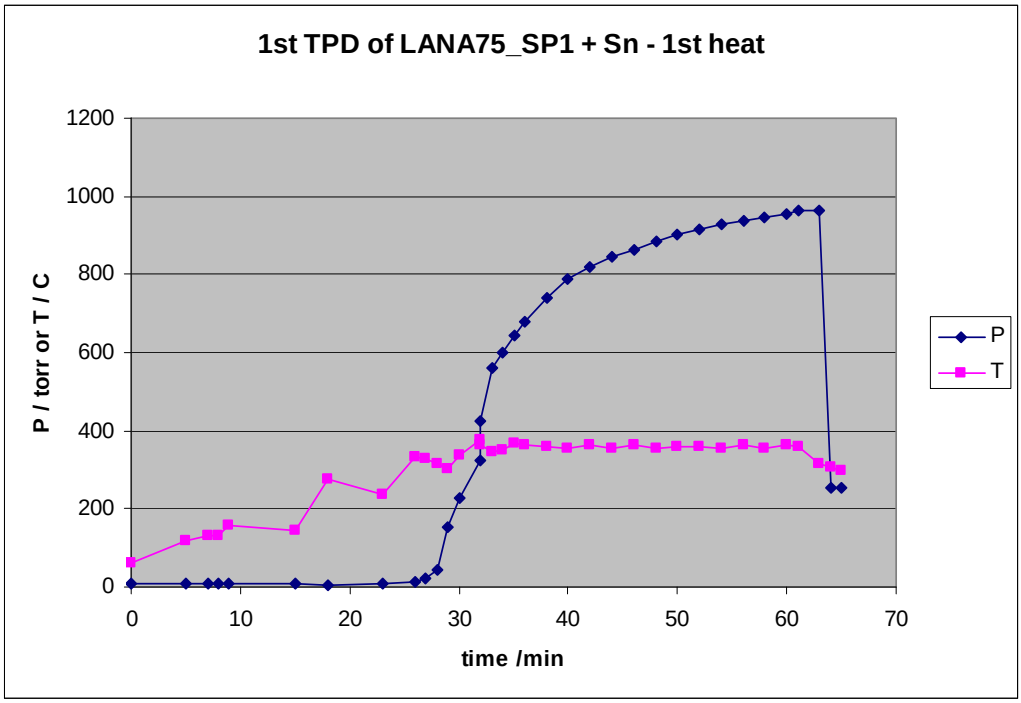

Figure 7. Pressure and Temperature profiles from 2nd heating of 1st Sn fusion sample

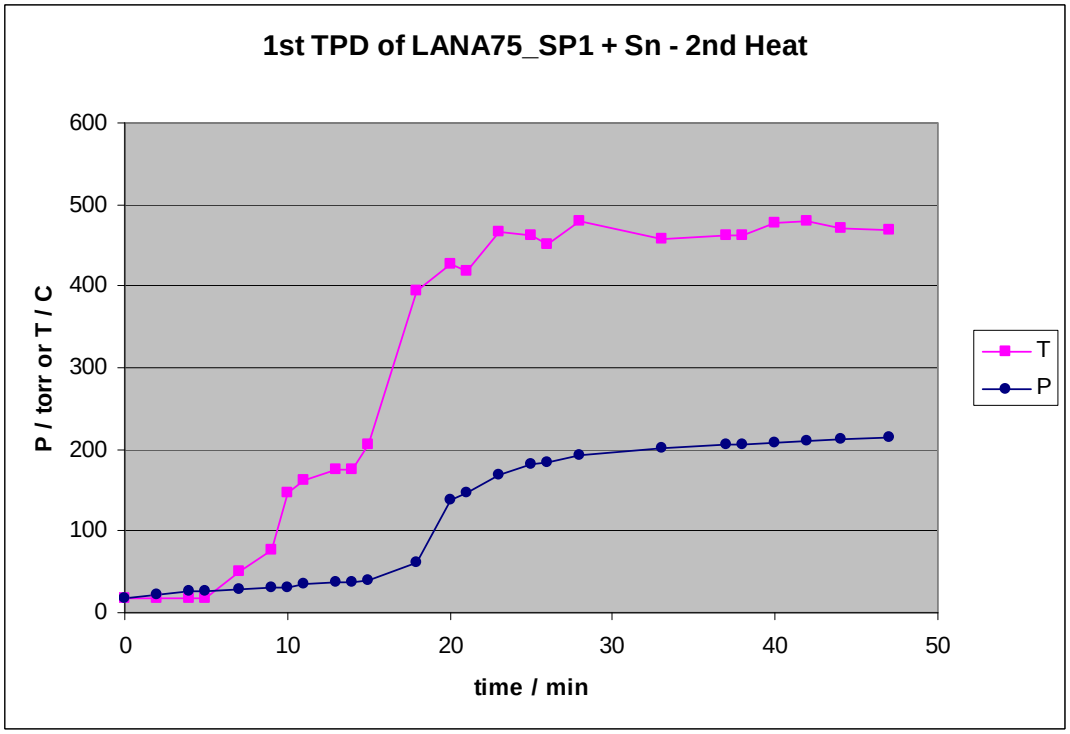

Figure 8. Pressure and Temperature profiles from 1st heating of 2nd Sn fusion sample

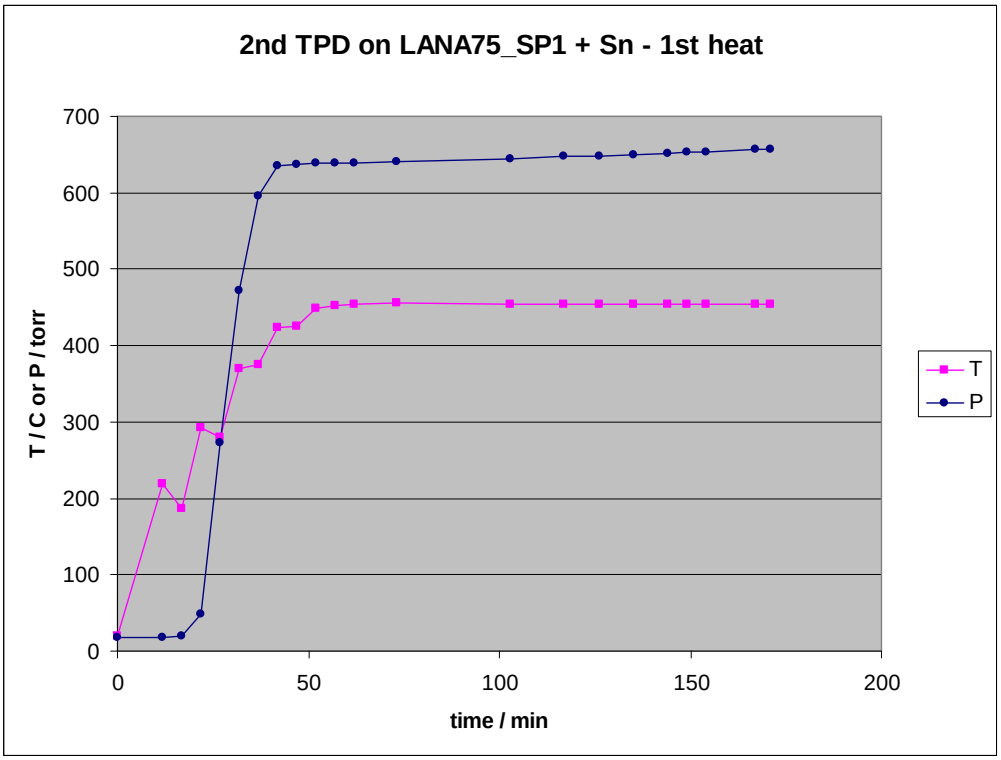
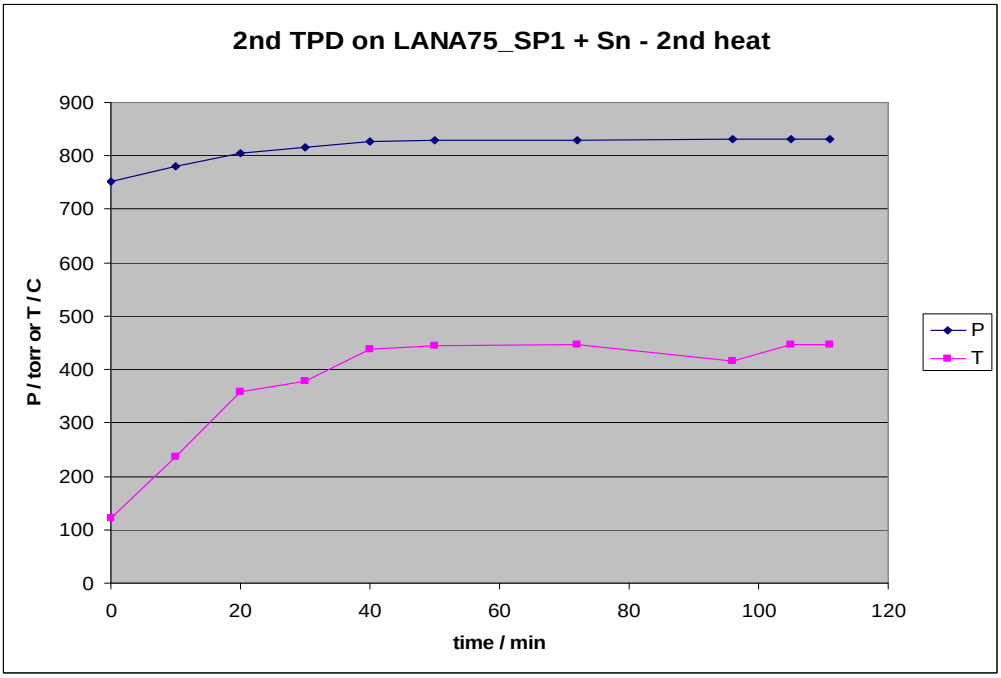
Figure 9. Pressure and Temperature profiles from 2nd heating of 2nd Sn fusion sample